

## **REMARKS**

The amendments to the above-captioned application along with the following remarks are being submitted as a full and complete response to the Office Action dated August 13, 2009. In view of the above amendments and the following remarks, the Examiner is respectfully requested to give due reconsideration to this application, to indicate the allowability of the claims, and to pass this case to issue.

### **I. Status of the Claims**

Claims 1 to 13 are pending in this application. Claim 10 to 13 have been withdrawn. Claim 1 has been amended to incorporate the limitations from original claim 9 and claim 9 has been cancelled from the application.

Claim 1 has also been amended to recite that both the polymer obtained by the polymerization and a purified polymer are granular. Support for this amendment can be found in the passages from lines 10 to 18 of the specification, and Examples 1 to 4, *inter alia*.

Further, claim 1 has been amended to change the proportion of water contained in the hydrophilic organic solvent from “1 to 30% by weight” to “1 to 20% by weight”. Support for this amendment can be found in the specification on page 23, lines 19 to 24, and Examples 1 to 4, *inter alia*. That is, the present specification describes that “If the content of water in the water-containing organic solvent used as the washing liquid is too high, the removal efficiency of a low molecular weight component (component extracted by Soxhlet extraction with chloroform) contained in the PAS may be lowered in some cases.” Specification, page 23, lines 19-24.

More particularly, Examples 1 to 4 of the specification show that washing a granular polymer with the hydrophilic organic solvents containing water in a proportion of at most 20% by weight is extremely effective to obtain a purified granular polymer.

### **II. Claim Rejection under 35 U.S.C. 103(a)**

In the Office Action, claims 1-9 were rejected under 35 U.S.C. 103(a) as being unpatentable over EP 0648797 or JP 2000-273175 or JP 04-139215. This rejection is traversed and reconsideration is respectfully requested.

The *KSR* Court reiterated that the framework for an objective analysis of obviousness under 35 U.S.C. 103 is stated in *Graham v. John Deere Co.* Obviousness is a question of law based on underlying factual inquiries. The factual inquiries enunciated by the *Graham* Court are: (i) ascertaining the scope and content of the prior art; (ii) ascertaining the differences between the claimed invention and the prior art; (iii) resolving the level of ordinary skill in the pertinent art; and (iv) evaluating secondary considerations. MPEP 2141.

In order to establish a *prima facie* case of obviousness, the Examiner must show each of the claim's limitations in combined references and that one of ordinary skill in the art would find the combination obvious or the Examiner must explain "why the difference(s) between the prior art and the claimed invention would have been obvious to one of ordinary skill in the art." MPEP 2141 (III).

First, these references do not disclose a process for producing a poly(arylene sulfide) (PAS) by polymerizing a sulfur source including an alkali metal hydrosulfide and a dihalo-aromatic compound in the presence of an alkali metal hydroxide in an organic amide solvent wherein the content of nitrogen impurities are extracted in an extract by a mixed solvent containing 40% by weight of acetonitrile and 60% by weight of water from the purified polymer is at most 50 ppm on the basis of the weight of the polymer. More particularly, these references neither teach nor suggest the production process of the poly(arylene sulfide) which is conducted by (1) a dehydration step of heating and reacting a mixture containing an organic amide solvent, an alkali metal hydrosulfide and the alkali metal hydroxide in a proportion of 0.95 to 1.05 mol per mol of the alkali metal hydrosulfide, (2) charging step of adding an alkali metal hydroxide and water to the mixture remaining in the system after the dehydration step, as needed, in such a manner that 1.00 to 1.09 mol of the alkali metal hydroxide and 0.5 to 2.0 mol of water are present per mol of a charged sulfur source including the alkali metal hydrosulfide, (3) a first-stage polymerization step of adding the dihalo-aromatic compound to the mixture to subject the sulfur source and the dihalo-aromatic compound to a polymerization reaction at a temperature of 170 to 270°C in the organic amide solvent, thereby forming a prepolymer that a conversion of the dihalo-aromatic compound is 50 to 98%, and (4) a second-stage polymerization step of controlling the amount of water in the reaction system after the first-stage polymerization step so as to bring about a state that water exists in a proportion of 2.0 to 10 mol per mol of the charged

sulfur source, and heating the reaction system at 245 to 290°C, thereby continuing the polymerization reaction.

Importantly, the cited references neither teach nor suggest that washing of a granular PAS polymer obtained by polymerization is conducted by sufficiently washing the granular polymer with the hydrophilic organic solvent containing water until the content of nitrogen contained in an extract extracted with the mixed solvent of acetonitrile/water becomes at most 50 ppm on the basis of the weight of the polymer.

The production process of the poly(arylene sulfide) by polymerizing a sulfur source and a dihalo-aromatic compound in an organic amide solvent has a tendency to produce a nitrogen atom-containing compound as a by-product. In particular, when a sulfur source and a dihalo-aromatic compound are polymerized in the presence of an alkali metal hydroxide in an organic amide solvent, the organic amide solvent such as N-methyl-2-pyrrolidone (NMP) is easily reacted with the alkali metal hydroxide to produce a compound containing a nitrogen atom as impurities under such an alkaline condition at high temperature. For example, when NMP reacts with sodium hydroxide (NaOH), NMP is subjected to ring opening to produce sodium methylaminobutanoate  $[(CH_3)NH-CH_2-CH_2-CH_2-COONa]$ . Sodium methylaminobutanoate reacts with the dihalo-aromatic compound that is a monomer component. For example, sodium methylaminobutanoate reacts with p-dichlorobenzene to produce sodium chlorophenylmethylaminobutanoate. Such a nitrogen atom-containing compound is difficult to be sufficiently removed even when the PAS is purified in a washing step after the polymerization. For example, even when the PAS obtained in the polymerization step is washed with an organic solvent or water, thereby removing by-products such as NaCl, and the content of a low-molecular weight component extracted by Soxhlet extraction with chloroform is reduced to at most 5% by weight, preferably at most 4% by weight, more preferably at most 3% by weight, the nitrogen atom-containing compound remains as impurities.

As a result, these impurities adhere to a mold or die when the PAS is injection-molded or extruded, presenting a problem. Since stain of the mold or die, which is caused by such a nitrogen atom-containing compound, adversely affects the quality of a molded or formed product, the mold or die requires frequent cleaning.

Applicant has carried out an extensive investigation to obtain a PAS reduced in the content of impurities composed of a nitrogen atom-containing compound. As a result, it has

been found that in a production process of a PAS by polymerizing a sulfur source including an alkali metal hydrosulfide and a dihalo-aromatic compound in the presence of an alkali metal hydroxide in an organic amide solvent, the content of impurities composed of a nitrogen atom-containing compound produced by the cause of decomposition of the organic amide solvent can be markedly reduced by washing the polymer obtained by the polymerization with a hydrophilic organic solvent containing water in a proportion of 1 to 20% by weight. The content of the impurities containing the nitrogen atom can be objectively evaluated by determining an amount of nitrogen contained in an extract extracted from the polymer by a mixed solvent containing 40% by weight of acetonitrile and 60% by weight of water.

It has been surprisingly found that the impurities composed of the nitrogen atom-containing compound can be markedly reduced in the case where the PAS formed is washed with the water-containing organic solvent without a significant effect on the content of a low-molecular weight component and this is so whether the PAS is washed with the water-containing organic solvent or by singly using the organic solvent or water as a washing liquid.

In addition, it has been found that PAS can be washed in the form of a granular polymer, and a purified PAS can be collected in the form of a granular polymer after washing.

As apparent from the results shown in Table 1 of Applicant's specification, it is understood that in the PASs of Comparative Examples 1 and 2 obtained by washing with a hydrophilic organic solvent, the content of the low-molecular weight component as determined by the Soxhlet extraction is considerably reduced, but the content of nitrogen derived from impurities remains at high level.

In the PAS of Comparative Example 3 obtained by washing with water, both of the content of the low-molecular weight component and the content of nitrogen derived from impurities are not substantially reduced and remain at high level.

In contrast, in the PASs (Examples 1 to 4) obtained by washing with a hydrophilic organic solvent containing water in a proportion of 1 to 20% by weight, the content of the low-molecular weight component as determined by the Soxhlet extraction is at the same level as or somewhat higher than the PASs of Comparative Examples 1 and 2 obtained by washing the organic solvent, but the content of nitrogen derived from impurities is markedly reduced. As a result, the PASs according to the present invention are markedly inhibited from volatilization of

impurities composed of a nitrogen- containing compound or adhesion to molding or forming apparatus thereof, which is caused by the impurities.

**A. EP 0648797 A2**

EP 0648797 A2 (EP reference) discloses a process for the production of a low-corrosive poly(arylene sulfide), in which a poly(arylene sulfide) is subjected to an organic acid treatment in a liquid mixture composed of an organic solvent and water in a weight ratio ranging from 4:1 to 1:10, and containing an organic acid in a concentration of 0.1-5.0 wt.%, and the thus-treated poly(arylene sulfide) is then thoroughly washed, is provided (abstract).

The EP reference does not disclose a process for producing a PAS by polymerizing a sulfur source and a dihalo-aromatic compound in the presence of an alkali metal hydroxide in an organic amide solvent. In addition, the sulfur source used in EP reference does not include an alkali metal hydrosulfide.

Further, unlike Applicant's invention, the EP reference does not disclose extraction of nitrogen impurities wherein the extract extracted by a mixed solvent containing 40% by weight of acetonitrile and 60% by weight of water from the purified polymer is at most 50 ppm on the basis of the weight of the polymer. The EP reference does not disclose removal of nitrogen impurities at all, let alone described by the specific characteristic that nitrogen in an extract extracted by a mixed solvent containing 40% by weight of acetonitrile and 60% by weight of water from the purified polymer is at most 50 ppm on the basis of the weight of the polymer. The EP reference discloses production of PAS high in crystallization but low in corrosiveness; it does not disclose removal of impurities as in Applicant's invention.

More importantly, EP reference neither teaches nor suggests that washing before an acid treatment should be conducted by sufficiently washing a polymer with the hydrophilic organic solvent containing water until the content of nitrogen contained in an extract extracted with the mixed solvent of acetonitrile/water becomes at most 50 ppm on the basis of the weight of the polymer.

EP reference describes that after the acid treatments, wet cakes containing PPS polymers were washed with water by two or three times (in all Examples). However, by washing with water, the content of the impurities composed of the nitrogen atom-containing compound cannot be substantially reduced and remains at high level.

EP reference neither teaches nor suggests that the acid treatment and washing after the acid treatment should be conducted until the content of nitrogen contained in an extract extracted with the mixed solvent of acetonitrile/water becomes at most 50 ppm on the basis of the weight of the polymer.

Further, nowhere in the EP reference does it teach or suggest the production process of the poly(arylene sulfide) which is conducted by (1) a dehydration step of heating and reacting a mixture containing an organic amide solvent, an alkali metal hydrosulfide and the alkali metal hydroxide in a proportion of 0.95 to 1.05 mol per mol of the alkali metal hydrosulfide, (2) charging step of adding an alkali metal hydroxide and water to the mixture remaining in the system after the dehydration step, as needed, in such a manner that 1.00 to 1.09 mol of the alkali metal hydroxide and 0.5 to 2.0 mol of water are present per mol of a charged sulfur source including the alkali metal hydrosulfide, (3) a first-stage polymerization step of adding the dihalo-aromatic compound to the mixture to subject the sulfur source and the dihalo-aromatic compound to a polymerization reaction at a temperature of 170 to 270°C in the organic amide solvent, thereby forming a prepolymer that a conversion of the dihalo-aromatic compound is 50 to 98%, and (4) a second-stage polymerization step of controlling the amount of water in the reaction system after the first-stage polymerization step so as to bring about a state that water exists in a proportion of 2.0 to 10 mol per mol of the charged sulfur source, and heating the reaction system at 245 to 290°C, thereby continuing the polymerization reaction.

In fact, the Examiner concedes that the references “do not disclose the specific steps of the process, as in claim 9.” *Office Action*, page 3. Applicant has amended claim 1 to specifically include these process steps. The Examiner argues that “these steps of the process are included in the broad teachings of the references. Therefore, it would have been obvious to one of ordinary skill in the art to select the steps from the references within the limitation of the instant claims since they have been shown to be effective in a similar system and thus would have been expected to provide adequate results. There is no showing of unexpected results derived from said selection.” *Id.* These conclusory statements fail to establish a *prima facie* case of obviousness. The Examiner has failed to explain *why* the difference between the prior art and Applicant’s invention would have been obvious to one of ordinary skill in the art. Mere conclusory statements that one of ordinary skill in the art would find it obvious to select these

steps is insufficient to establish a *prima facie* case of obviousness, yet that is exactly what the Examiner has done in the Office Action.

**B. JP 2000-273175**

JP 2000-273175 discloses a purification process of a poly(arylene sulfide) resin, in which a poly(arylene sulfide) resin is washed in a softened or molten state with a mixed solvent of an aprotic organic solvent and water by means of a line mixer (claim 1).

The purification process of JP 2000-273175 is a purification process for efficiently removing by-produced alkali halides such as lithium halide remaining in the PAS resin. In the purification process of JP 2000-273175, the washing temperature is set to a range of 220 to 320°C to soften or melt the PAS resin. In the purification process of JP 2000-273175, the washing of the PAS resin was performed in a washing time of less than 3 minutes and at a washing temperature of 220 to 320°C using the line mixer ([0012]).

More specifically, Example 1 of JP 2000-273175 shows that a melted polymer phase was extracted from the bottom of a reactor after a polymerization reaction, and this polymer phase was mixed with a mixed solvent of NMP and water and introduced into a static mixer. The static mixer was adjusted to give an internal temperature of 270 to 275°C. The residence time of the mixture in the static mixer was about 10 seconds. A washing process by such a static mixer was conducted 4 times. It is shown that after the washing, a PAS resin in the form of cake was taken out, ground and vacuum-dried.

JP 2000-273175 discloses an alkali metal sulfide such as lithium sulfide as a sulfur source. However, JP 2000-273175 neither discloses nor suggests using a sulfur source containing an alkali metal hydrosulfide.

In addition, the purification process disclosed in JP 2000-273175 is markedly different from the washing step adopted in the present invention. That is, according to the present invention, the washing of the polymer obtained by polymerization is generally conducted, after separating a product slurry by filtration to collect a granular polymer, by sufficiently washing the resulting filter cake comprising the granular polymer with the hydrophilic organic solvent containing water until the content of nitrogen contained in an extract extracted with the mixed solvent of acetonitrile/water is at most 50 ppm on the basis of the weight of the polymer.

In the purification process of JP 2000-273175, the washing is conducted at a high temperature and in an extremely short period of time (typically, about 10 seconds) in the static

mixer, so that if nitrogen atom-containing impurities are contained in the PAS resin, it is presumed that the impurities cannot be sufficiently washed out and removed. According to the purification process of JP 2000-273175, deterioration of the PAS resin is easily advanced under high-temperature purification conditions, and heat fusion bonding between PAS resins is caused to proceed. In order to obtain powdered polymer, it is required to pulverize the fused polymer.

In contrast, in the washing step according to the present invention, PAS can be washed as it is in the form of a granular polymer, and the purified PAS can be collected in the form of a granular polymer after washing. In addition, according to the washing step of the present invention, the polymer can be sufficiently washed until the content of nitrogen contained in an extract extracted with the mixed solvent of acetonitrile/water is at most 50 ppm on the basis of the weight of the polymer.

#### **C. JP 4-139215**

JP 4-139215 discloses a washing process of a poly(arylene sulfide), comprising, upon washing of poly(arylene sulfide) particles obtained by reacting an alkali metal sulfide and/or an alkali metal hydrosulfide with a dihalo-aromatic compound in a polar solvent and separated from a reaction mixture, washing the poly(arylene sulfide) particles with an organic solvent having a boiling point lower than water and compatibility with the above-described polar solvent and water, and then washing the thus-washed particles with water (claim 1).

JP 4-139215 also discloses a washing process comprising drying the poly(arylene sulfide) particles separated from the reaction mixture and then successively conducting washing with an organic solvent and washing with water (claim 2).

JP 4-139215 further discloses a washing process comprising drying the poly(arylene sulfide) particles separated from the reaction mixture and then washing the particles with a mixed solvent of an organic solvent and water (claim 3).

JP 4-139215 does not disclose a process for producing a PAS by polymerizing a sulfur source and a dihalo-aromatic compound in the presence of an alkali metal hydroxide in an organic amide solvent.

That is, JP 4-139215 describes, regarding the production process of PAS, that

“A necessary amount of an alkali metal sulfide or alkali metal hydrosulfide and optionally a polymerization aid are first added into a desired polar solvent, a dehydration operation is conducted by azeotropic distillation, vacuum distillation or the like according to circumstances, a



necessary amount of a dihalo-aromatic compound and various kinds of additive components optionally used are then added, and the resultant mixture is heated to a temperature in a range of generally 180 to 330°C, preferably 220 to 300°C to conduct a polymerization reaction.” (Page 5, left upper column, lines 7-16).

There is no disclosure in JP 4-139215 relating to a process for producing a PAS by polymerizing a sulfur source and a dihalo-aromatic compound in the presence of an alkali metal hydroxide in an organic amide solvent.

In the washing process disclosed in JP 4-139215, the poly(arylene sulfide) particles separated from the reaction mixture are dried and then washed in case of washing with the mixed solvent of the organic solvent and water.

Specifically, Examples 4 to 14 of JP 4-139215 show experimental examples that dried PAS particles were washed with a mixed solvent of methanol/water (1/1), acetone/water (1/1), ethanol/water (1/1), or isopropyl alcohol/water (1/1) (Table 1 at page 9, upper right column). The contents of water in these mixed solvents are all 50% by weight.

JP 4-139215 does not specifically disclose washing with the hydrophilic organic solvent containing water in a proportion of 1 to 20% by weight. When the content of water in the water-containing organic solvent used as the washing liquid is too high, the removal efficiency of a nitrogen atom-containing compound is lowered.

In addition, according to the present invention, washing process may be conducted, after separating a product slurry by filtration to collect a granular polymer, by washing the resulting filter cake comprising the granular polymer with the hydrophilic organic solvent containing water until the content of nitrogen contained in an extract extracted with the mixed solvent of acetonitrile/water is at most 50 ppm on the basis of the weight of the polymer. After washing step, the wet cake containing the granular polymer is dried. Therefore, it is not required to dry the polymer before washing with the hydrophilic organic solvent containing water.

There is no disclosure in these references relating to the selected combination of constituent features defined by claim 1. There is no suggestion, express or implied, in these references relating to possibility of achieving further improvement, particularly in extremely reducing the nitrogen atom-containing compound by combining such teachings along the line of the present invention.

In view of the foregoing, the EP reference, JP 2000-273175, and JP 4-139215 fail to render claim 1 obvious under a proper 103 analysis as one skilled in the art cannot predictably arrive at the claimed invention by following these references' teachings and suggestions. As with claim 1, claims 2-9 are similarly considered by Applicant to patentably define themselves over these references by virtue of their dependency from claim 1.

It is therefore submitted that the presently claimed invention recited by claims 1-9 are nonobvious over and patentably distinguishable from the cited references taken individually or in combination, and that the rejection under 35 U.S.C. 103(a) has been overcome. Reconsideration is respectfully requested.

### **III. Conclusion**

In light of the above-outlined Amendments and Remarks, Applicants respectfully request early and favorable action with regard to the present application, and a Notice of Allowance for all pending claims 1 to 8 is earnestly solicited.

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Respectfully Submitted,



Nanda P.B.A. Kumar  
Registration No. 44,853  
Jenny Papatolis  
Registration No. 61,284  
Reed Smith LLP  
2500 One Liberty Place  
1650 Market Street  
Philadelphia, PA 19103-7301  
(215) 241-7991  
Attorneys for Applicant